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October 18, 1991

In reply refer to G.O. 71033

Office of Naval Research
800 No. Quincy Street
Arlington, VA 22217-5000

Attention: Dr. Wallace A. Smith
Subject: Quarterly R&D Status Report No. 4
"Electrodeposition of High Temperature Superconductors"
For period 07/01/91 through 09/30/91
Contract No. N00014-90-C-0225
SC71033.QRDSR

Enclosed is subject report.

ROCKWELL INTERNATIONAL CORP.
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15 October 1991 In reply refer to G.O. 71033

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800 North Quincy Street
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Statement A per telecon Dr. Wallace Smith
ONR/Code 1131
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NWW 11/15/91

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PROGRAM SUMMARY

The overall objective of this project is to develop a process for direct electrodeposition of Y-Ba-Cu superconducting oxides from a molten salt at relatively low temperatures (300-550° C). The approach entails establishing a sequence of electrochemical steps for the layered deposition of Y, Ba and Cu oxide species from a eutectic Na-K nitrate melt.

PROGRAM STATUS

The background data needed to define appropriate procedures/voltage sequences for electrodeposition of Y-Ba-Cu HTSC oxides have been obtained. Electrodeposition of CuO has been investigated in detail and shown to be insensitive to temperature (at least to 400° C). All three metals have been demonstrated to electrodeposit from the nitrate melt and the current-voltage characteristics for the deposition/dissolution processes have been established. Both Cu and Y have been shown to electrodissolve in the melt and to deposit as the oxides (CuO and Y_2O_3). Deposition of Y oxide on Cu oxide electrodes results in a uniform film composition over a 0.4 μm thicknesses, indicating that formation of mixed metal oxide compounds occurs. Since direct oxide electrodeposition occurs (at least for Cu and Y) in the nitrate melt, this system is ideally suited for deposition of HTSC materials. Direct metal oxide deposition presumably involves reduction of nitrate complexed with the metal cation and therefore should be a general phenomenon applicable to preparation of a wide range of mixed metal oxides.

ACCOMPLISHMENTS

Background studies needed for design of appropriate HTSC deposition schemes were completed this quarter. All three metals have been demonstrated to electrodeposit from the nitrate melt and the current-voltage characteristics for the deposition/dissolution processes have been established. Figure 1 gives voltammograms showing deposition and dissolution of each metal oxide at a rotating Pt disk electrode. The deposition onset potentials are: 0.4 V vs Ag/Ag^+ for Cu; -0.1 V for Y; and -1.3 V for Ba. Both Cu and Y metals have been shown to electrodissolve in the melt. Figure 2 shows that temperature has little effect on the CuO deposition process up to at least 400°C.

Based on x-ray diffraction (XRD) and Auger electron spectroscopy (AES) data for deposits, both Cu and Y deposit as the oxides (CuO and Y_2O_3) from the nitrate melt. This presumably occurs via reduction of nitrate (complexed with the metal ion) to nitrite as illustrated for CuO deposition by:

$$Cu^{2+} + NO_3^- + 2e^- \dashrightarrow CuO + NO_2^-$$

Such a reaction mechanism suggests that it may be possible to deposit a wide variety of metal oxides from the molten nitrate system.

PROBLEM AREAS

None.

GOALS FOR NEXT REPORTING PERIOD

Future work will focus on developing and evaluating promising schemes for electrodeposition of Y-Ba-Cu HTSC materials. Initial studies will be directed toward evaluating the molten salt electrochemical equivalent of molecular beam epitaxy. In this case, the electrode voltage is maintained just positive of that required for Ba oxide deposition, and monolayer amounts of Cu and Y are injected (by electrodissolution of individual metal electrodes) and electrodeposited in sequence. A cell of very small volume is used to ensure that complete deposition of the injected metal occurs in a short time. Incorporation of Ba oxide layers should occur in proper sequence by underpotential compound formation. This simple straightforward approach will be investigated thoroughly before more complicated deposition schemes are considered.

Rockwell International Science Center

D. M. Tench

D. M. Tench
Principal Investigator

FIGURES

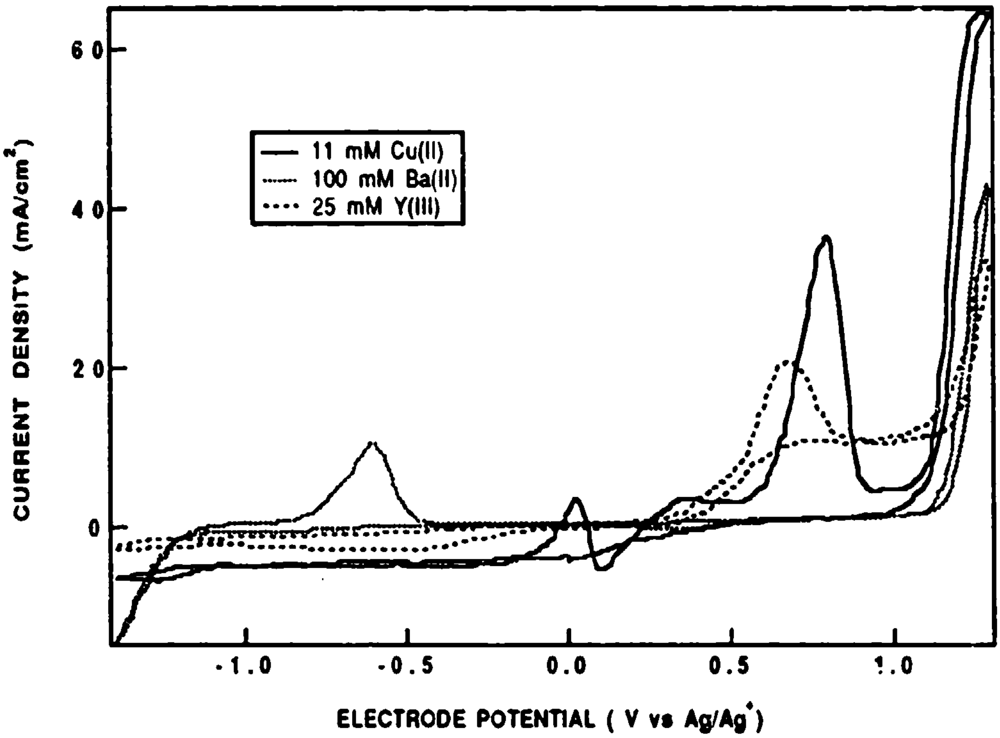

Fig. 1 Linear sweep cyclic voltammograms (300 mV/s) for a Pt rotating disk electrode (600 rpm) in the eutectic Na-K nitrate melt (300°C) containing Cu(II), Y(III) or Ba(II) species.

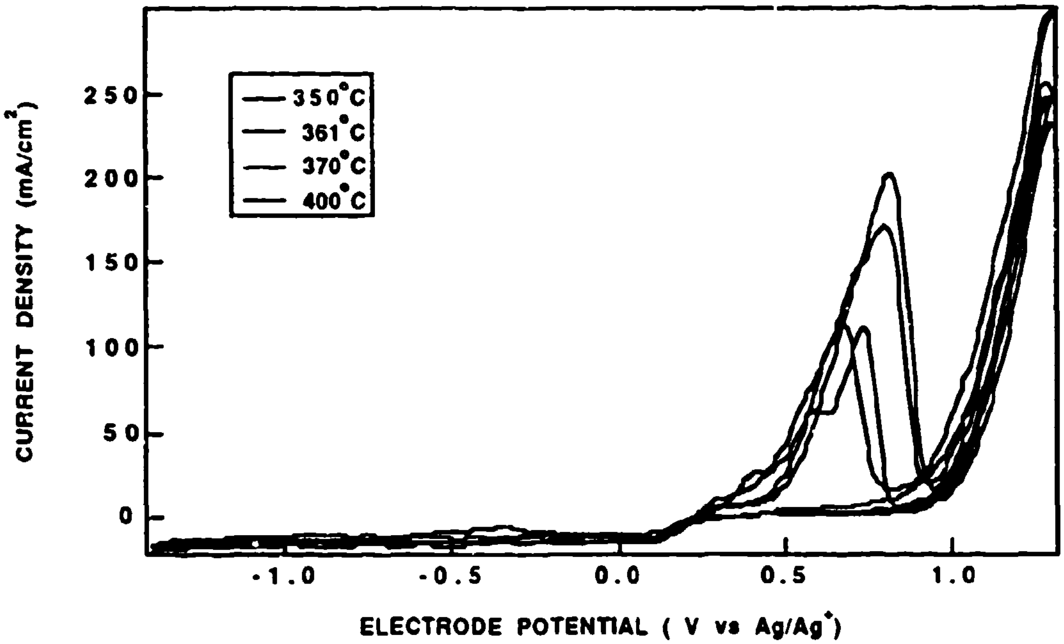

Fig. 2 Effect of temperature on CuO deposition and dissolution at a Pt rotating disk electrode (conditions same as for Fig. 1)